Precipitate Formation in the Distribution System Following Addition of Orthophosphate

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Introduction

Orthophosphate is being used by many water utilities as a metal corrosion inhibitor. The substance binds with metal ions, creating a film on the outer pipe scale to prevent corrosion. After the Washington Aqueduct (WA), the District of Columbia Water and Sewer Authority's (WASA) finished water supplier changed disinfection from free chlorine to chloramines, lead leaching accelerated in the water distribution system. WA began to add phosphoric acid in response to the reported higher lead levels. The initial target residual was 3.2 mg/L phosphate, which WASA intended to maintain until the corrosion control treatment optimized. The 90th percentile lead levels declined from 59 to 15 ppb within eight months of phosphate addition.

WASA began to receive customer complaints of white cloudy water after a year of continuous phosphate treatment. Water Quality technicians collected and tested water samples from taps and hydrants in response to these complaints. Several hydrant samples had a white cloudy appearance and color_{apparent} exceeding 200 Pt-Co units. Significant higher levels of phosphate, iron, and aluminum were also measured in these hydrant samples. Filtering the hydrant samples showed nearly all the iron and aluminum and a majority of the phosphate were in particulate form. Interestingly, technicians rarely captured this cloudy appearance inside tap samples. WASA conducted a study, involving field and bench-scale analyses, to determine the cause of the white cloudy water.

Theory

In distribution systems, aluminum, calcium, ferrous and ferric ions can react with various anions and negative-charged organic matter to form precipitates, complex ions and/or coagulate, which may lead to flocculation (Kemmer et al; Miller et al; Lente et al). All mechanisms are observed in most systems. The degree in which one mechanism predominates over another depends on all other cations and anions present, pH, alkalinity, and temperature (Lytle et al).

WA's addition of phosphate into the water supply increased the potential for phosphate to influence particulate formation. Past research has shown that phosphate can release minerals from corrosion layers, decrease bacterial adhesion, form precipitates, and adsorb onto metal oxides, hydroxides, and/or other products (Appenzeller et al; Lytle et al; Magnuson et al; EPA, 2000).

The following paragraphs outline some theoretical particulates and colloids formed from aluminum, calcium and iron; which are known to increase color_{apparent} readings and tend to give a white cloudy appearance. In each section, phosphate particulate formation with each metal is discussed.

Aluminum Formations Aluminum precipitation and coagulation are important reactions within wastewater and drinking water treatment. Aluminum salt addition to raw water and wastewater allows aluminum to react with phosphates to form aluminum phosphate and aluminum hydroxides (Haas et al). When enough aluminum salt is added to raw water to neutralize the negatively charged particulates and colloids present, coagulation begins. If the appropriate mixing energy is then applied, flocculation proceeds. The flocs formed are later removed, however this removal is not absolute and a small amount of aluminum salts usually leaves the treatment plant. The formations of crystalline aluminum phosphate and aluminum hydroxide solids are competing reactions and can be given the generalized formula Al(PO₄)₃(OH)_{3-x} (Haas et al). In low phosphate concentrations (<10ppm) the competition is more

significant and will lead to an amorphous collection of particulates and colloids (Yeoman et al.) The amount of amorphous particulates and colloids produced depends on phosphate concentration and pH.

Calcium Formations Calcium is usually the major metal present in semi-hard to hard water distribution systems. Particulate formation is often influenced and/or involved with this cation. The primary precipitate is the crystalline solid, calcium carbonate. Langlier Saturation Index is often used to characterize water's tendency to form this solid. This formula relates temperature, moles of calcium, pH and alkalinity to the formation of calcium carbonate.

Independent of calcium carbonate formation is the precipitation of calcium phosphate particulates or colloids by the formation of apatites (e.g. $CaHPO_4$; $Ca_4H(PO_4)_3$; $Ca_3(PO_4)_2$) or hydroxyl apatite ($Ca_5(PO_4)_3OH$ (Haas et al). This particulate is usually seen in higher pH ranges around 9 and once formed can re-dissolve, if pH is around 7.0 and phosphorus concentration is less than 3ppm (Haas et al).

Iron Formations Ferric and ferrous ions are known to form many particulates and deposits within distribution systems (i.e. goethite, magnetite, lepidocrocite, ferrihyrdrite) (Vikesland et al). An oxidant used for microbiological disinfection in distribution systems will oxidize most ferrous ions in solution to ferric ions. This oxidation reaction is autocatalytic and is also accelerated by the presence and/or formation of iron oxides. The kinetics of ferrous ion oxidation and influence of dissolved oxygen, a weak oxidant, will influence the physical characteristics of ferric particulates formed.

Once ferric ions are formed, they will react with alkalinity and/or phosphate to form insoluble precipitates. These particulates can be generalized by the equation:

$$wPO_4{}^{3\text{-}} + xFe^{3\text{+}} + yOH^\text{-} \\ \longleftrightarrow Fe_x(PO_4)_w(OH)_y{}^{(3x\text{-}3w\text{-}y)}$$

Iron phosphate and iron hydroxide formation are competing reactions and are dependent on pH, alkalinity, and phosphate concentration. If more ferric than phosphate ions are present the formation of ferric hydr(oxide) and positively charged complex ions will predominate. Conversely, if more phosphate ions than ferric ions are present the formation of ferric phosphates and negatively-charged ions will predominate. This process, unlike coagulation (mechanism that would proceed if more ferric hydroxides are formed), allows for the formation of negatively charged particles. These products will repel each other thereby making it easy to re-suspend and/or keep them in suspension.

Sources of Al, Ca, Iron, and phosphate in WASA's distribution system

The sources of metal cations and anions within a distribution system can be found in the source water, treatment chemicals added at the plant, and pipe and pipe-scale dissolution. WASA's distribution system iron sources are limited to the piping dissolution and corrosion layer deposits, since no iron salts are used in drinking water treatment. These two sources are significant considering the high percentage of cast iron piping in the distribution system. For aluminum, typical sources are alum and poly-aluminum chloride added at the water treatment plant. Calcium sources are more varied than the other metals, since calcium is found in natural raw water, added at the treatment plant in the form of lime, and incorporated in pipe scale layers. Phosphate is primarily from the phosphoric acid addition at the treatment plant.

This paper outlines the investigation of the white cloudy water phenomena within WASA's distribution system. WASA conducted field analyses to determine the extent of the problem and gather current data. Further bench-scale experiments were performed to characterize the physical properties of the aluminum, calcium, iron, and phosphate interactions under WASA's typical conditions. Bench-scale particulate volume measuring and settling tests were also performed. X-ray diffraction with computer modeling of laboratory formulated and field precipitates are presented to further describe, compare, and identify the major elemental species that contribute to this white cloudy water.

Procedures

Field Sampling

A field sampling plan was developed from three distribution system zones¹ that incorporated white cloudy characteristics. Within two zones, the distribution location with the highest color_{apparent} sample was targeted for Scanning Electron Microscopy (SEM) and Electron Detector System (EDS) analysis conducted by the University of Washington. The sampling method was to collect a sample from a hydrant after flushing for 15 minutes at a rate of 250 gpm. Flushing at this flow rate and time allows for cleaning of the hydrant line while also collecting loose debris in the main. Individual constituents were analyzed using HACH methods. Correlation attempts were then made to determine each constituent's effect on apparent color.

Scanning Electron Microscopy (SEM) was employed in an attempt to identify any distinct mineralogical or crystallographic forms within the precipitate mass captured in the field sampling program. The solid precipitates were captured and concentrated on nitro cellulose filters (0.45 micron). This analysis was conducted at the University of Washington using a JEOL 4800 LV SEM system with an EDAX module.. In order to enhance the imaging capability of the instrument and avoid surface charging, the filtered specimens were sputter coated with gold. However, even given this precaution it was impossible to identify any distinct morphologic forms within the precipitate filter cake. The macroscopic character of the collected particles was largely indistinguishable, consisting entirely of amorphous (non-crystalline) particulate matter.

The Energy Dispersive X-ray Spectrometer (EDS) analysis was conducted utilizing a diffractometer producing Copper K-alpha (K_a) radiation controlled by manufacturer supplied software. This analytical technique could successfully determine both the elemental atomic distribution of the filtered precipitates, as well as respective elemental mass distribution.

Bench Scale Testing

Bench-scale experiments evaluated metal salt formations within typical WASA distribution water. Aluminum, calcium, and iron interactions from typical to elevated levels² of phosphate were investigated with several batch experiments. Each batch reactor consisted of a 1000 ml beaker, a variable speed mixer, and stir bar. Stock solutions of orthophosphate, aluminum, calcium, and iron were prepared from 60% phosphoric acid, aluminum sulfate solution, calcium chloride solid and ferric chloride solid, respectively.

The tap water used for all experiments was drawn from the entry point containing approximately 3.2 mg/L of phosphate as PO₄. Four batch reactions were conducted, each batch containing a different constituents; aluminum, calcium, iron and phosphate. Each constituent was added at incremental concentrations to the batch reactors. Lime or potassium hydroxide was added for pH adjustment when necessary to maintain a pH of 7.5, equivalent to average pH levels found in WASA's distribution system.

After each chemical addition, the mixture was stirred at 10 rpm for 2 minutes, then reduced to 2 rpm to minimize floc breakup. Slow mixing continued for approximately 30 minutes. Samples were drawn while the mixture was stirring. Color_{apparents} pH, and the relevant metals were measured.

All water samples were analyzed using a HACH 890 colorimeter, reagents, and procedures. Each reactor was observed and analyzed after each metal addition and at a final 24-hour retention time.

¹ Zones are grouped by water usage and pressure within the distribution system

² These residual level amount was taken from the review of historical data of distribution system and identifying the highest value recorded.

Additionally, a separate series of experiments are presented under the section: <u>Evaluation of Iron Phosphate Formation</u>. This series of experiments was performed to visually evaluate ferric particulate formation under three conditions; de-ionized water, tap water, and tap water plus elevated phosphate

Experimental overview

Scenario I. Evaluation of Aluminum Phosphate interaction in color apparent development

Aluminum addition

Incremental doses of 0.1 ppm of Al³⁺ to reach a maximum cumulative dose of 0.6 ppm were added to entry point tap water with a phosphate residual of 3.5 mg/L as PO₄.

Scenario II: Evaluation of Calcium Phosphate interaction in color apparent development

Calcium addition

The Langlier Saturation Index (LSI) of the distribution system water was calculated, to maintain the LSI value below the threshold value for which calcium carbonate precipitation begins. Therefore, Calcium chloride solution was added at 25 ppm Ca²⁺ per increment, and the final value was 213 ppm Ca²⁺. Additions of HCl and KOH solutions were added to keep pH at approximately 7.6.

Scenario III: Evaluation of Iron Phosphate interaction in colorapparent development

1. Iron addition

Ferric chloride was added in increments of 0.20 ppm Fe⁺³ to a final cumulative concentration of 1.88 ppm. Orthophosphate was kept at constant value of 3.5 ppm. To correct the drop in pH, lime solution was added to increase the pH to 7.5. To account for the lime imparting color, a lime blank was developed.

2. Constant Fe³⁺ ion concentrations in three environment

HACH Iron standard solution (ferrous sulfate dissolved in an acidic medium) was added to three 300 ml glass reactors—two with distribution system water and one with deionized water. The final iron concentration for each reactor was approximately 2.30 mg/L. Phosphate standard solution was added to increase the concentration of orthophosphate to 11.8 mg/L. To oxidize ferrous ion to ferric ion, 2 ml of sodium hypochlorite (15%) (1200 mg/L) was added to the mixture. The pH of the mixture was maintained at 7.0 – 8.0 with addition of potassium hydroxide.

The mixing of this experiment differed from the others. The mixture was stirred at 8 rpm for 2 minutes, then reduced to 2 rpm for approximately 30 minutes. The mixture was measured for iron, phosphate, pH, and color_{apparent} readings, after a 24-hour reaction period.

Table 1. Summary of various batch experiments scenarios

Scenario	Experiment	Al, mg/L	Fe, mg/L	PO4, mg/L	Ca, mg/L
I. Aluminum-	1.Orthophosphate	0.406	_	3.2 - 16.0	
Phosphate	addition				
interaction	2.Aluminum	0 - 0.6	-	3.2	-
	addition				
II.	1.Orthophosphate	-	-	2.8 - 6.8	175
Calcium-	addition				
Phosphate	2.Calcium	-	-	3.8	113 - 238
Interaction	addition				
III.	1.Iron addition	-	0.1 - 1.88	3.5	-
Iron-phosphate	2.Phosphate	-	2.3	3.5 - 11.8	-
Interaction	addition				

Table 2. Initial tap water quality

PO4, mg/L	Al, mg/L	Color	Fe, mg/L	рН	Temp
3.2	0.000	0	0.02	7.56	18.9

Floc Volume Measurements

Additional bench-scale experiments were conducted to quantify the particulate or floc formed under varying phosphate doses and fixed concentration of iron, aluminum, and calcium. An Imhoff cone was used to quantify the volume of precipitate formed. Floc volume was measured in mL/L at intervals of 24, 40, 46, and 106 hours. A summary of metal and phosphate concentrations added is located in Table 3.

Experiment 1 Floc volume measured due to ferric-phosphate interaction

Ferric chloride was added to each beaker, producing 2.0-mg/L ferric. Phosphate addition varied from 2.6 to 20.0 mg/L among the five beakers.

Experiment 2. Floc volume measured due to aluminum -ferric-phosphate interaction

The same procedure was followed as in experiment 1 with the addition of 0.7 mg/L aluminum.

Experiment 3. Floc volume measured due to calcium -ferric-phosphate interaction.

The same procedure was followed as experiment 1, with the addition of 230 mg/L calcium.

Table 3. Summary of experimental conditions in measuring floc volume

Experiment	Fe,	PO4, mg/L	Al, mg/L	Ca, mg/L	Time intervals in floc
	mg/L				measurement, hrs
Ferric-	2.0	2.6 - 20.0	-	-	24, 40, 46 & 106
phosphate					
interaction					
Aluminum -	2.0	2.6 - 20.0	0.7	-	24, 40, 46 & 106
ferric-					
phosphate					
interaction					
Calcium -ferric-	2.0	2.6-20.0	-	230	24, 40, 46 & 106
phosphate					
interaction					

RESULTS AND DISCUSSION

FIELD DATA ANALYSIS AND INTERPRETATION

Scanning Electron Microscope (SEM) and Electron Detector System (EDS) analysis of particulates from distribution system with cloudy water

Cloudy water samples collected from the two highest apparent color locations in the distribution system were filtered with 0.45 micron filter paper. The collected particulates were analyzed using SEM method. The spectra of the constituent elements are tabulated in Tables 4 and 5 and Figures 1.1 and 1.2.

Table 4. Percentage composition of precipitates of cloudy water in distribution 1

Distribution	Element	N	F	Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe	Cu
1 (H1)														
Spectra	Wt %	6.53	4.11	1.08	6.50	6.11	1.77	6.13	0.79	0.72	0.54	49.69	14.74	0.61
	At%	15.23	7.07	1.54	8.74	7.40	2.06	6.47	0.80	0.67	0.45	40.52	8.63	0.31

Table 5: Percentage composition of precipitates of cloudy water in distribution 2

Distribution 2 (H2)	Element	N	F	Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe	Cu	Pb
Spectra	Wt %	5.91	5.20	0.00	8.47	9.27	1.43	8.45	0.41	0.28	0.23	50.51	8.49	0.86	0.49
	At%	13.32	8.64	0.00	11.00	10.85	1.61	8.62	0.41	0.25	0.18	39.81	4.8	0.43	0.08

Fig 1.1 Percentage composition of particulates in distribution zone 1

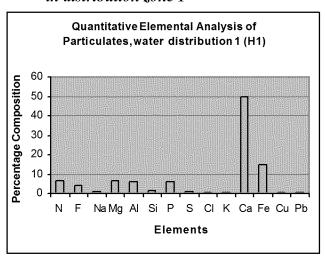
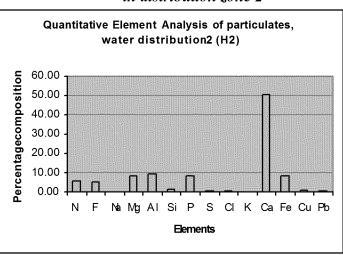


Fig 1.2. Percentage composition of particulates in distribution zone 2



As shown in the graphs, the percentage composition of the water samples from two distribution sites is very similar. The elemental percentage composition of the particulates shows the predominance of calcium (40% –50%); moreover, aluminum, iron, and phosphorus constitute a major part of the particulates.

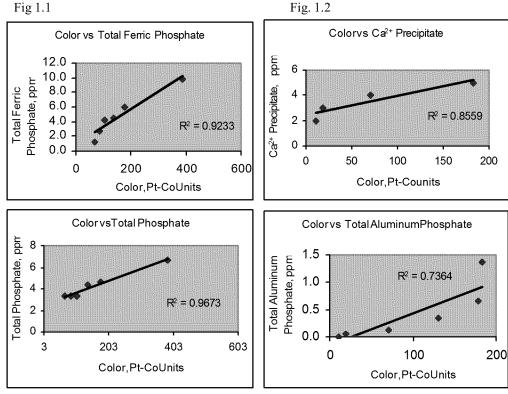
Individual Constituents vs. Apparent Color

WASA collected and analyzed hydrant samples from three different zones in the distribution system where white cloudy water was previously observed. All field samples collected for this analysis had a white cloudy appearance. Data were analyzed by area (or zone) to show if various phosphate compounds versus color_{apparent} relationships differ within the distribution system and if an individual constituent had a strong correlation.

Aluminum and iron measured in the analyzed zones are expressed in the following figures as total aluminum phosphate and total iron phosphate to demonstrate metal phosphate particulate. This was done based on empirical formulas and the assumption that the metal reacted with the phosphate and was the limiting reactant. Calcium precipitate is the difference between total Ca²⁺ value measured in solution and Ca²⁺ value in the filtrate.

Distribution Zone #1

As iron, aluminum, phosphate and calcium precipitate increased, color_{apparent} was observed to increase.



Distribution Zone #2



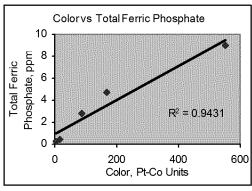
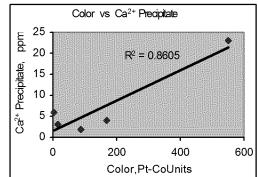
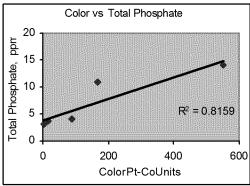


Fig 2.2





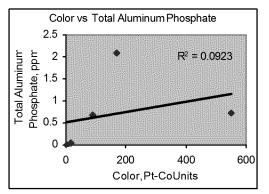


Fig 2.3

Fig 2.4

Distribution Zone #3

Fig 3.1

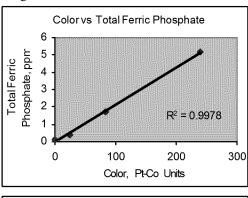
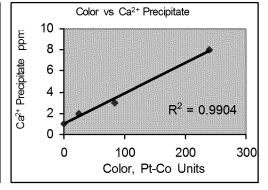
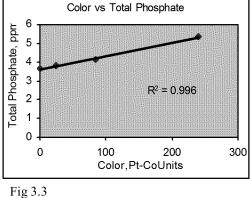


Fig 3.2





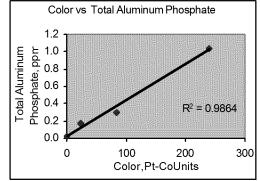


Fig. 3.4

In all the hydrant zones, the correlation between color_{apparent} and phosphate was consistently strong. In addition, ferric and calcium were similar to phosphate R-squared values. This suggests that these two metals may be the major constituents that interact with phosphate to form a precipitate. Aluminum also appears to increase as color increases although the correlation is much weaker.

Overall, average trends appear to indicate that these metal's interaction with phosphate are all involved in cloudy color development.

Bench Scale Experimental Results

I. Results of Metal Phosphate Batch Experimentation

Scenario I. Aluminum and phosphate interaction in particulate formation

Aluminum addition to water with 3.2 mg/L orthophosphate

Figure 4 shows that as phosphate increases, color_{apparent} increases, though the linear regression value is only 0.782. Color_{apparent} values were extremely low in the bench scale test compared to Color_{apparent} values found in the distribution zones

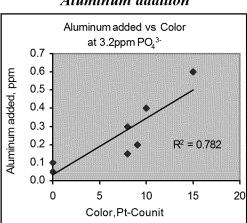


Figure 4.
Aluminum addition

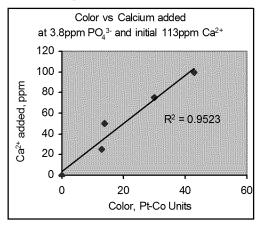
Scenario II. Calcium and phosphate interaction in color formation

Calcium addition to water with phosphate levels of 3.8 mg/L

Figure 5 displays a maximum Color_{apparent} value of 43 Pt-Co units with Color_{apparent} values increasing as calcium hardness increases. This graph illustrates similar trends compared to the distribution analysis conducted in Figures 1.2, 2.2 and 3.2, in which, color_{apparent} values appear to be a function of calcium concentration.

Figure 5.

Calcium addition



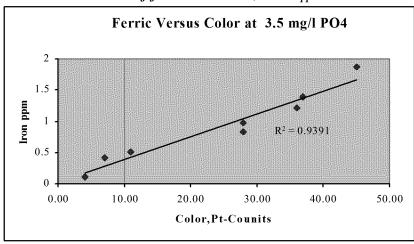
Observations of batch reactors after the 24hr-holding period showed almost no particles had settled.

Scenario III: Iron addition to tap water with phosphate

1. Iron addition to water with 3.5 mg/L orthophosphate

This experiment was done under controlled pH. The pH of the water was maintained around 7.5 with the addition of lime. It was observed that cloudy flocs were formed, and the results show that color_{apparent} progressively rose as the concentration of iron increased.

Figure 6.
As the concentration of ferric increases, color_{apparent} increases.



In harmony with field data of cloudy water from the distribution system, this bench scale experiment further shows that iron-phosphate interaction could cause formations of particulates to make the water cloudy. Moreover, noticeable amount of particles of a light brown hue settled at the bottom.

2. Constant Fe³⁺ ion concentrations in three environment conditions

To further investigate iron particulate formation, a bench scale experiment was conducted for iron under three conditions (see Table 6). This experiment showed a change of particulate appearance when phosphate levels were changed. Experiment A is a ferric oxide particulate

and experiment C is predominately ferric phosphate. These results suggest that changing phosphate concentration changes particulate appearance.

Table 6. Appearance of particulates changes as the concentration of phosphate changes

Experiment	A	В	C
	De-ionized water +	Tap water+	Tap Water + Iron +
Conditions	Iron	Iron	Phosphate
Ferric Iron, mg/L	2.29	2.30	2.21
PO ₄ , mg/L	0.07	3.50	11.80
Ferrous (dissolved)			
Iron mg/L	0.03	0.02	0.08
PO ₄ , dissolved, mg/L	0.06	1.02	1.45
color _{apparent} Pt-Co	81	57	79
Particulate phosphate	0	70	87.7
%			
Total Chlorine -	0	3.2 mg/L	3.2 mg/L
Chloramines			_
Visual observations	Orange brown	light brown	White cloudy

Experiment A (DI +iron) produced colloids, brown in color most likely ferric hydroxide. However, experiment B (3.5 mg/L phosphate + iron) produced colloids that were lighter brown than colloids formed in experiment A. Analysis of filtered and collected particulates shows that 2.48 mg/L of phosphate (70%) and almost all the iron were incorporated within. In experiment C particulates were cloudier than in experiments A or B. In Experiment C, out of 11.8 mg/L total phosphate only 1.45 mg/L was dissolved, the remaining 87.7 % of the phosphate was in particulate form, and most of the iron (96.4%) was in particulate form. This shows that the flocs, which are cloudy, are mainly composed of iron-phosphate complexes.

II. BENCH SCALE EXPERIMENTS IN MEASURING FLOC VOLUME

Results from floc volume measuring

1. Ferric- Phosphate Interaction and floc formation

It was observed that flocs formed and developed due to the interaction of ferric with phosphate. The sludge volume measured by the Imhoff cone increased as the phosphate concentration increased. Moreover, the floc volume increased as contact time increases indicating that noticeable particulate formation is likely to occur further out near the ends of the distribution system (Figures 7.1 and 7.2).

Fig 7.1 Floc volume increases as phosphate increases at 2.0 mg/L Fe

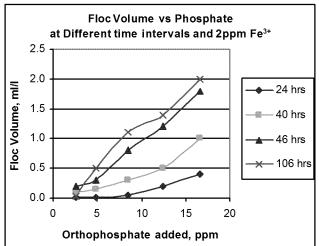
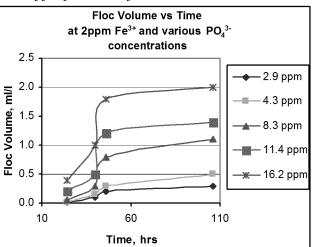


Fig 7.2
Rate of floc formation is fastest at 24-48 hrs.



It was further observed that the rate of floc formation increased after 24 hours. Initially it takes time for the nucleus of floc to be formed; however once the seed of flocs is formed, the rate of floc growth is relatively fast. The floc formation is considerably higher after 24 hrs contact time and there is no considerable increase in floc formation after 48 hrs of contact time, as can be seen in Fig 7.2.

2. Aluminum-Ferric-Phosphate interaction and floc formation

The introduction of 0.7 mg/L aluminum resulted in an increase in floc volume compared with the floc volume formed by interaction of only ferric-phosphate. Aluminum may play a role in enhancing ferric-phosphate interaction in floc formation.

Fig 8.1 Floc volume increases as PO_4 increases at 0.7 ppm Al and 2.0 ppm Fe

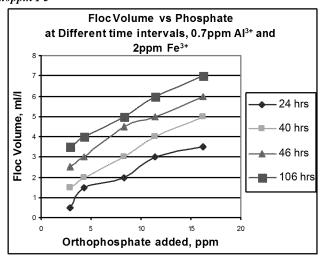
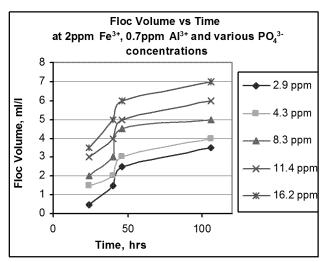


Fig 8.2
Rate of floc formation is fastest 24-48 hrs



The rate of floc formation is similar as in ferric-phosphate interaction. Floc formation is considerably high at 24 - 48 hrs of contact time.

3. Calcium-Ferric-Phosphate interaction and floc formation

Similar to the ferric-phosphate experiment, floc formation increased over time producing similar floc volumes. However, increasing concentrations of phosphate seem to have little effect on sludge development in the presence of calcium. This was unexpected and yielded different results compared to batch experiment Scenario II where mixing calcium and phosphate yielded increasing color. However, color_{apparent} is not a true measurement of precipitation formation and is not related to the amount of settled precipitate as measured in the Imhoff apparatus. High color and low settled floc volumes may indicate that the floc that formed under the influence of calcium has a tendency to remain in suspension.

Fig 9.1
Floc volume was the same as phosphate increased at 230ppm Ca and 2.0ppm Fe

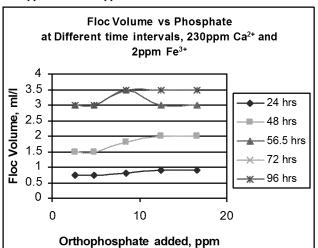
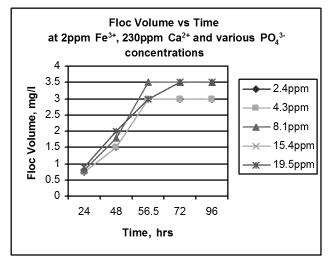


Fig 9.2
Rate of floc formation is fastest at 48-60 hrs



Conclusions

- High levels of phosphate (3.2 mg/L) in the presence of metal ions formed flocs in the distribution system, which produces the white cloudy appearance. Major constituents found in the particulate consisted primarily of phosphorous, iron, calcium, magnesium and aluminum by weight. However, based on phosphate's greater affinity for iron oxides than all other inorganic or organic anions (Appenzeller et al), it maybe assumed that iron-phosphate interaction plays a more pronounced role in flocs produced in the distribution system than the other metals studied.
- Other metal ions within the distribution can be incorporated within the iron phosphate flocs, influencing particulate characteristics.
- Aluminum may increase floc volume in the presence of ferric-phosphate, although it was not definitive from the data.
- Formation of precipitation in the presence of high calcium and iron concentrations may not be dependent on phosphate concentrations as shown in Figure 9.1. However, Figures 5 and 6 show a direct correlation of increased apparent color as each constituent's concentration was increased in the presence of phosphate, but in the absence of the other constituent.
- Formation of particulate phosphate can be complex in drinking water thus indicating the need for
 further study with phosphate-metals reactions, particulate formation and characteristics and volume
 of precipitate formed.

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